US PTO 2000-1306 SU 1717679

Translated from the Russian

U.S.S.R.
State Committee
for Inventions and Discoveries at the
State Committee of the U.S.S.R. for Science and Technology
Description of Invention
with Author's Certificate
SU 1717679 A1
IPC: D 06 P 1/28
Date of application: April 13, 1990
Date of making available to the public the claims only of the Description of Invention with Author's certificate: March 7, 1992, Bulletin No. 9
Applicant: Moscow Textile Institute named after A. N. Kosygin
Inventors: A. G. Timatkov et al.
Prior art documents:

Krichevskiiy, G. E. et al., Khimicheskaya tekhnologiya tekstil'nykh materialov [Chemical technology of textile materials]- Moscow, Publishers: Legprombytizdat, 1985, p 462.

Belen'kiiy, L.I. et al., Trudy TsNIKhBI [Transactions of Central Scientific and Research Institute of the Cotton Industry], Moscow, 1958.

Gt. Britain Pat No. 1259530, Class D 18 2 E, 1972.

U.S.S.R. Author's Certificate No. 1513057, D 06 P 1/28, 1989

Gt. Britain Pat. No, 1499402, Class D 1B 2E, 1978

Andreeva, M.V., Krashenie smesey polyefirnykh volokon s natural'nymi [Dyeing of mixtures of polyester fibers with natural ones]

METHOD FOR THE COLORING OF TEXTILE MATERIALS

(57) The invention pertains to the dyeing-and-finishing industry, in particular to the dyeing of textile materials by means of vat dye-sols. The invention provides an opportunity for the technology to be simplified (to reduce the amount of dyeing solution, to reduce its flow-rate, to eliminate the dye-fixing stage), to expand the range of textile materials, which can be

colored (providing thus an opportunity not only for the dyeing of cotton and cotton, containing polyester ingredients, but for the dyeing of viscose, linen, silk, wool, fabrics of polyvinyl alcohol fibers, as well as of fabrics, made of mixtures of cotton with polyurethane) as well as to increase the intensity of their coloring or dyeing by 12 to 33% while the strength parameters of the coloring remain the same. The said positive effect is achieved on account of the fact that the fabric is treated with an aqueous solution, containing vat dye-sol and sodium dithionite or sodium hydrosulfite, accompanied by subsequent irradiation by means of a source of light, having a maximal emission in the spectrum, having a wavelength of 250 to 400 nm [i.e 200 to 400×10^{-9}m] and an energy of emission or radiation equal to 0.2 to 0.3 Joules/cm, for a period of 2 to 4 minutes. 2 Tables.

FIRST TRANSLATOR'S NOTE: The inventors & applicant are using all over the text of this patent document the Russian term "kubozol'". It is a term, which is related but different from the term "kubovoy krasitel'" = vat dye(stuff)= vat color [in English]= Küpenfarbstoff [in German]. Now let us analyze the term "kubozol'" which is a combination of the words "kub" + "zol'" with an "o" in the middle for acoustic reasons in Russian. First, the Grand English - Russian Technical Dictionary specifies that: "kub" = 1. vat;
= 2. solution of a reductive (regenerative) form of a vat dyestuff {Küpenfarbstoff in German]. The plural in Russian is "kuby" = sodium salts of reduction (deoxidation) products, and that
"zol" = sol = a liquid colloidal dispersion.
Now, in the case when we have vat dyestuff ["kubovoy krasitel'" in Russian] or vat dyestuffs, everything is clear, as far as coining of the term is concerned. But in the case of a "sol of a solution of a reductive (regenerative) form of a vat dyestuff", the Russian inventors have coined the single-word term "kubosol'" [vat-sol" by substituting the group of words "solution of a reductive (regenerative) form of a vat dyestuff" with the word "vat" and forming the word "kubozol", or, literally, in English,

a vat-sol [U.K.] or vat sol. It is suggested already by Derwent, that in order to keep the continuity and to indicate that the sol pertains to the solution of a vat dyestuff we retain the words "vat dyestuff". Thus, finally, it is suggested that "kubozol'" is translated as vat-dyestuff sol wherein it is implicit that when we say 'vat dyestuff'. It is to be implicitly understood that the point in question is not the vat dyestuff but a solution of a reductive (regenerative) form of a vat dyestuff. Thus, the word 'vat-dyestuff sol' or 'vat dyestuff sol' will be used in this translation.
SECOND TRANSLATOR'S NOTE: Due to space and time considerations, the tables will not be constructed again in a computer-aided way but only the contents of the tables (more than 100 entries) will be translated. Please, see original tables for additional numeration or reference symbols of the entries.

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The invention pertains to the chemical technology, in particular to the dyeing or coloring of textile materials by means of vat dyestuff sols.

A nitride method for the dyeing of textile materials by means of vat dyestuff sols is known, according to which method the fabric is immersed in a solution of vat dyestuff sol whereby the ratio of the components is as follows, in g/l: vat dyestuff sol 1 to 2; soda ash 1; sodium nitrite 8; moistening agent 1. The temperature of the solution is 40 to 50°C. After the processing, the fabric is squeezed, treated in a solution of sulfuric acid, having a concentration of 30 g/l at a temperature of 70 to 80°C, followed by rinsing in two tubs by means of cold and, after that, by hot water (75 to 80°C). After this, the processing is done in a solution, containing 5 g/l of a 40% oleic soap and 2 g/l soda

ash. at 60 °C, and the rinsing is done in three tubs by means of hot water (75 to 80°C) followed by drying.

However, in the process of development [of the dye], deleterious nitrogen oxides are discharged, it is difficult to obtain an intensive coloring, and an overoxidation of the various brands of vat dyestuff sols is possible, which leads to a blunted coloration whereby the acid medium contributes to a rapid corrosion of the equipment.

Another method for the dyeing by means of vat dyestuff sols is known, which involves the use of γ-radiation, including the impregnation of the textile material with an aqueous solution of vat dyestuff sol at 60°C for a period of 30 seconds whereby the ratio of the components of the dye vat in g/l is: vast dyestuff sol 10; sulfuric acid 5, sodium nitrite 80. The soaked or impregnated fabric specimens are squeezed out, and subjected to the irradiation by means of radioactive isotope of cobalt, cobalt-60. The radiation dose [exposure] is 1 to 5 Mr [sic]. After this, the fabric is rinsed with running water, processed for 5 minutes in a boiling solution of oleic soap, having a concentration of 2 g/l.

However, that method requires intricate equipment and a strict system for the protection of the personnel from the effect of ionizing radiation.

Another method of dyeing by means of vat dyestuff sols is known, involving their subsequent development by means of a light source. The dye solution contains, besides vat dyestuff sol,

acetic acid, having a hydrogen-ion concentration,pH, up to 4 to 5. The rinsing composition contains, in g/l,: surface active substances 5; sodium carbonate 2; polyvinyl pyrrolidone 1 to 2. The development of dye takes place by means of luminescent, xenon, or fluorescent lamps.

The disadvantages of that known method consist in the high flow-rate of chemical reagents over the course of rinsing. The acid medium of the dyeing solution has an adverse effect on the strength properties of the fabric, which are being processed, and leads to corrosion of the equipment. Besides this, the method does not provide for the achievement of the additional coloristic effects of the photographic printing [photocopying].

Another method for the dyeing of textile materials by means of vat dyestuff sols is known, which method involves treatment of the textile material by means of a composition, containing, in g/l: vat dyestuff sol 1 to 10; polyvinyl acetate emulsion (50%-emulsion) 1 to 30; water up to 1 l,
and subsequent irradiation of the impregnated material by means of a source of ultraviolet light (mercury vapor lamp)for a period of 1.5 to 5 min. In order for a printed picture or pattern to be produced by using the composition in question, 40 to 60 g/l of thickening agent are introduced into the said composition, and the textile material is printed through a meshy or latticed template or stencil. No rinsing is carried out.

The disadvantage of that known method consists in the increase to some extent of the hardness of the touch [hand of

fabric]. Also, the method does not provide for any effects, inherent to the photographic printing.

Another method for the photographic coloring of cotton and mixed (cotton/polyester) fabric is known, which method involves impregnation by means of an aqueous solution, containing, in g/l: vat dyestuff sol 18; ammonium sulfate 8 to 9; ammonium vanadate 0.1 to 0.2; ammonium thiocyanate (or ammonium dichlomate [sic!]) 2. The dye solution is prepared immediately prior to the impregnation in a preset sequence. The impregnated material is squeezed out, and passed through a chamber, containing a light source (a xenon lamp). After this, the fabric is spaced in a [dye-] fixing solution of soda, having a concentration of 2 g/l, and is aged for a period of 24 hours, followed by two times of boiling in a relevant solution and in water for a period of 30 minutes, followed by drying. The method provides an opportunity to color textile materials in an intensive color, and to attain certain coloristic effects of the photographic printing.

However, that known method has a long duration, is intricate in its technological embodiment, and requires preparation of multicomponent solutions immediately before their use as a result of which the process becomes more complicated. The high flow rate of chemical reagents renders the processing more expensive, and complicates the wastewater treatment. Besides this, the use of a mere xenon lamp narrows the spectrum range of the vat dyestuff sols, which can be developed in a photochemical way, and prolongs the processing time. A limiting factor is also the insufficiently

wide range or assortment of the textile materials, which are being processed, which are colored in accordance with the proposed method.

It is an object of the invention to expand the variety of the textile materials, which can be colored in accordance with the proposed method.

The objective thus set is achieved as a result of the fact that in accordance with the proposed method for the coloring of textile materials, involving processing of the material by means of a composition on the basis of a vat dyestuff sol, followed by irradiation with a source of ultraviolet light, a solution of vat dyestuff sol and reducing agent in water is used in its capacity as a composition for impregnation whereby the ratios of the ingredients are as follows, in g/l: vat dyestuff sol 1 to 15; sodium dithionite or sodium hydrosulfite 5 to 10; water up to 1 l, and as source of ultraviolet light is used as used in its capacity as source of radiation (mercury-vapor lamp; excimer [-laser?] lamp; xenon lamp, ultraviolet laser), which has the maxima of the emission in the range of the spectrum, having wave lengths of 250 to 400 nm, and an energy of radiation of 0.2 to 0.3 Joules/cm^2. The irradiation is carried by means of a negative or without such. Sodium dithionite or sodium hydrosulfite is used as an additive & reducing agent. The reducing agents contribute

to the development of the vat dyestuff sols, exposed to a concurrent action of ultraviolet light.

The reduction of the concentration of the reducing agent (less than 5 g/l) leads to a deterioration of the coloristic characteristics of the dyed fabric, and to a counterproductive expenditure of vat dyestuff sol and electric energy, whereby the increase (more than 10 g/l) complicates the process of wastewater treatment.

As the wavelength of the effective light [to which the product is exposed] diminishes (less than 250 nm), the instrumentation [equipment] management of the process becomes more intricate, and there arises a possibility of a damage to the fabric. As the wavelength increases (more than 400 nm), the retention time necessary for the irradiation increases by 5 to 20 times, which leads to a counterproductive electric energy input.

The method thus proposed does not require a long-term fixing of the dyeing, preparation of intricate multi-ingredient solutions and cumbersome equipment or apparatuses. The solutions used are stable in storage.

The textile material (e.g., cotton, linen fabric, materials made of viscose, polyvinyl alcohol fibers, natural silk, wool, of mixtures of cellulose fibers with polyester and polyurethane fibers) is dipped in water solution, containing, in g/l: vat dyestuff sol 1 to 15; sodium dithionite or sodium hydrosulfite 5 to 10; water up to 1 l.

After this, there follows squeezing out up to 75 to 125% gain in weight, and irradiation with a source of UV light, having maxima of emission in the domain of 250 to 400 nm, and a capacity of 0.20 to 0.30 Joules/cm^2, for a period of 2 to 4 minutes through a negative or without it from a distance of 150 to 350 m. After this, the textile material is rinsed with hot water, a boiling solution of surface active substances (OP-10, sintanol, prevocell [*Translator's note: Most probably, the last two words are trademarks of the now defunct Soviet Union, but no further information is available in our databases]), having a concentration of 2 to 5 g/l for a period of 10 min, followed by rinsing with cold water, and drying.

The parameters of the method are tabulated in Tables 1 and 2.

In accordance with the method, 30 g of vat dyestuff sol (anthrasol-colors, made by "Hoechst", Germany) [German name: Anthrasol-Farbstoffe, Hoehcst GmbH] are dissolved in 600 g of boiling water, 40 g of a solution of ammonium sulfate, prepared by means of dissolving 1 part by mass of ammonium sulfate in 2 parts of hot water, 20 g of a solution of ammonium vanadate, prepared by dissolving 1 part of ammonium vanadate in 100 parts of hot water, 10 g of an ammonium thiocyanate solution, prepared by dissolving 1 part by mass of thiocyanate in 2 parts of hot water, are mixed with the solution of vat dyestuff sol and 1,000 parts of cold water.

After the impregnation, the fabric is squeezed out, subjected to irradiation by a lamp, having a power of 5 KW, neutralized in a soda solution, having a concentration of 2 g/l, for a period of 24 hours, after which boiling is carried out for a period of 30 minutes.

C L A I M

Method for the coloration of textile materials, by means of treating the material with a composition, containing vat dyestuff sol and an additive, and subjecting the impregnated material to an irradiation by means of a source of light, c h a r a c t e r i z e d in that in order for the technology [technological process] to be simplified and the range of assortment of the textile materials to be colored to be expanded while the intensity of their dyeing is concurrently increased and its stability is maintained, sodium dithionite or sodium hydrosulfite area, used as an additive, whereby the ratio of the ingredients in g/l is as follows: vat dyestuff sol 1 to 15; sodium dithionite or sodium hydrosulfite 5 to 10; water up to 1 l followed by irradiation by means of a light source, having maxima of emission in the range of the spectrum, which has a wavelength range from 250 to 400 nm and an energy of irradiation [irradiation capacity] of 0.20 to 0.30 Joules/cm^2) for a period of 2 to 4 minutes through a negative or without resorting to it.

L E G E N D

Symbols used in Tables 1 and 2. (Please, see Translator's note at the beginning).

TABLE 1.

The head of the table contains the following data.
Column I [Please, see the Roman numerals additionally added in the Russian text (Table 1) with red ink]:
Column I = Experiment #
II = Textile material [fabric]
III = Reducing agent
IV = Concentration of the reducing agent, g/l
V = Concentration of the vat dyestuff sol, g/l
VI = Exposure time, min
VII = Wavelength, nm [$1x10^{-9}m$]
VIII = Irradiation energy, Joules/cm^2
IX = Vat dyestuff sol-brand
X = Intensity of dyeing, K/S [unit unknown, not deciphered by the inventor]
XI = Strength characteristics of the dyeing (coloring) when subject to mechanical and chemical effect, in scale numbers
XI A = Resistance to treatment by soap
XI B = Resistance to distilled water
XI C = Resistance to friction
XI C1 = Resistance to dry friction
XI C2 = Resistance to moist [wet] friction
XI D = Resistance to light [photostability]
The numbers in column I are as follows:
1 = cotton fabric; [the relevant characteristic in column IX being bright pink Ж]
2 = fabric of viscose fiber; [column IX - brand - blue]
3 = linen fabric; [column IX - brand - red-brown, Ж]
4 = mixed fabric, i.e. cotton + polyester fabric [column IX - grey C]
5 = mixed fabric, i.e. viscose + polyester[column IX- Light Blue K]
6 = natural silk [column IX - Blue]
7 fabric made of polyvinyl alcohol fibers [Blue}
8 = knitted fabric [jersey] made of cotton [Column IX -Blue + bright pink Ж]
9 = knitted fabric of a mixture of cotton and polyurethane [column IX - Golden yellow ЖX]

TABLE 2
VERTICAL COLUMN
COLUMN 1 or A = Experiment #
COLUMN 2 or B = Impregnating composition and concentration of the reagents, g/l

COLUMN 3 or C = Required power [capacity], kW at a velocity , m/min
COLUMN 4 or D = Textile material
COLUMN 5 or E = Technique, employed for the development of the dye, irradiation time in min, energy [power] characteristics of the light source.
COLUMN 6 or F = Coefficient of Intensity of the dye, K/S [abbreviation not provided by the inventor]
COLUMN 7 or G = Strength of dye
COLUMN G1 = Resistance or strength to dry friction
COLUMN G2 = " to moist [wet] friction

Vertical column A or 1 i.e. EXPERIMENT #:
KNOWN Method
Experiment #1, involving in column B or 2,
a) = vat dyestuff sol, Blue
b) Ammonium sulfate
c) Ammonium vanadate
d) Ammonium thiocyanate
e) Water
PROPOSED Method Experiments ## 2 & 3, involving in column B,
a) Vat dyestuff sol, Blue
b) Sodium dithionite
c) Water
d) Vat dyestuff sol, Blue
e) Sodium Dithionite
f) Water
KNOWN Method, Experiment # 4, involving in column B,
a) Vat dyestuff sol, Gray C
b) Ammonium sulfate
c) Ammonium vanadate
d) Ammonium thiocyanate
e) Water
PROPOSED Method, Exp. # 5, involving in column B,
a) Vat dyestuff sol, Grey C
b) Sodium hydrosulfite
c) Water
PROPOSED Method, Exp. #6, involving in column B,
a) Vat dyestuff sol, Grey C
b) Sodium sulfide
c) Water
KNOWN Method, Exp. #7, involving in column B,
a) Vat dyestuff sol, Red-brown X
b) Ammonium sulfate
c) Ammonium vanadate
d) Ammonium Thiocyanate
PROPOSED Method, Exp. # 8
a) Vat dyestuff sol, Red-brown X
b) Sodium dithionite
c) Water

Exp. # 9,
a) Vat dyestuff sol, Red-brown X
b) Sodium dithionite
c) Water
KNOWN Method, Exp. #10,
a) Vat dyestuff sol, Blue
b) Ammonium sulfate
c) Ammonium vanadate
d) Ammonium thiocyanate
e) Water
PROPOSED Method, Exp. # 11
a) Vat dyestuff sol
b) Sodium hydrosulfite
c) Water

Exp. # 12* - (* = Change of the lamp (i.e. employing another wave characteristic of the light, to which the product is subjected.)
a) Vat dyestuff sol, Blue
b) Sodium hydrosulfide
c) Water
KNOWN Meth., Exp. # 13
a) Vat dyestuff sol
b) Ammonium sulfate
c) Ammonium vanadate
e) Water
PROPOSED Method**, Experiment # 14 , (** = Change of the irradiation time [dose of radiation].)
a)

Exp. # 15

a) Vat dyestuff, Blue
b) Sodium dithionite
c) Water

Exp. # 16

a) Vat dyestuff, Blue
b) Sodium dithionite
c) Water

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** For these symbols, see Experiments # 12 and # 14, 15, 16

US PATENT & TRADEMARK OFFICE
Translations Branch
January 20, 2000
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